# KINETICS OF POTASSIUM CATALYZED GASIFICATION

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INTRODUCTION

The Exxon Catalytic Coal Gasification Process[2] is based upon a new combination of processing steps which avoid thermodynamic constraints inherent in the previous art. The use of the catalyst in the reaction step and the manner in which the reactor is integrated into the overall process are the keys to this concept. The goal of the work reported here is the formulation of a kinetic relationship for catalytic gasification which can be used in developing a model for the fluid bed reactor used in this process. This model is needed to correlate pilot unit conversion data and as a design tool for commercial scale units. This paper reports on the work which culminated in the successful formulation of the required kinetic expression.

Alkali metal gasification catalysts increase the rate of steam gasification[3,4,5] promote gas phase methanation equilibrium,[2,5] and minimize agglomeration of caking coals.[1] The catalytic gasification process uses an alkali metal gasification catalyst (K_2CO_3) with a novel processing sequence which maximizes the benefits of the catalyst. The process combines a relatively low gasifier temperature (1300°F) and high pressure (500 psig) with the separation of syngas ($CO + H_2$) from the methane product. The syngas is recycled to the gasifier so that the only net products from gasification are CH_4, CO_2, and small quantities of H_2S and NH_3. The resulting overall gasification reaction can be represented as follows:

$$Coal + H_2O \rightarrow CH_4 + CO_2$$
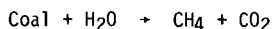
Since this reaction is essentially thermoneutral, major heat input to the gasifier at high temperature is not required. Thus, as discussed by Nahas and Gallagher[5], second law constraints on thermal efficiency inherent in other processes are avoided.

A simplified flow plan for the process is shown in Figure 1. Coal is impregnated with catalyst, dried and fed via a lockhopper system to a fluidized bed gasifier which operates at about 1300°F and 500 psig. The coal is gasified with a mixture of steam and recycled syngas. The major gasifier effluents are CH_4, CO_2, CO, H_2, and unconverted steam. No tars or oils are produced. The gaseous products are cooled and the unconverted steam is condensed. The dry product gas is treated in a series of separation steps including acid gas scrubbing to remove CO_2 and H_2S, and cryogenic fractionation to separate methane from syngas. The syngas is combined with feed steam and recycled to the gasifier at approximately 150°F above the gasification temperature. Although there is no net heat required for the gasification reactions, some small amount of heat input is required to heat up the feed coal, to vaporize residual water, and to provide for gasifier heat losses.

Ash/char residue from the gasification step is sent to a catalyst recovery unit in which a large fraction of the catalyst is leached from the residue using countercurrent water washing. The recovered catalyst, along with some makeup catalyst, is reimpregnated on the coal to complete the catalyst recovery loop.

EXPERIMENTAL

Design of the gasifier for this process requires a quantitative description of the kinetics of the catalytic gasification reaction. Bench scale studies were conducted in a fixed bed reactor to provide the necessary data for the development of the rate equation.

Apparatus

The high pressure apparatus used in this study is shown in Figure 2. The main components of the system are the fixed bed reactor, water pump and steam generation equipment, pressure and temperature control systems, unreacted steam condenser, a gas chromatograph and a dry gas flow measurement system. Provisions were included for the optional use of an inert or reactant gas (such as H_2 + CO) as a feed supplementing steam.

A high pressure pump was used to supply H_2O at a constant rate to the steam generator which consisted of 1/4" stainless steel tubing coiled around the fixed bed reactor. Both the steam generator and the reactor were mounted vertically in a split tube furnace. The reactor temperature was measured and controlled at the center of the bed of char. The product gas stream, consisting primarily of H_2, CO, CH_4, CO_2 and unreacted H_2O, was filtered and then depressurized through the pressure control valve. The unreacted H_2O was condensed and the gas stream was further dried by calcium sulfate. The dry gas stream passed through a gas chromatograph sampling system, which provided automatic sampling at 15-minute intervals. The dry gas flow was measured by a wet test meter connected to a pulse generator. The signals from the pulse generator were accumulated as a measure of total gas volume produced.

The fixed bed reactor was constructed from 1-inch Schedule 80 stainless steel pipe and was approximately 30 inches in length. The reactor was filled to a depth of 15 inches by 1/8-inch mullite beads which supported the bed of char.

Procedure

Samples were prepared by soaking 30 to 100 mesh Illinois coal No. 6 in a solution containing the desired weight of catalyst, typically between 10 and 20 gms K_2CO_3/100 gms of coal (referred to as 10 and 20% K_2CO_3). Normally, the weight ratio of water to coal was slightly greater than one. The samples were then dried overnight in a vacuum oven. A scanning electron microscope study showed a fairly even dispersion of potassium throughout the coal particle. The impregnated coal samples were then devolatilized at atmospheric pressure for 30 minutes in a muffle furnace under a nitrogen atmosphere at 1200°F. The samples were allowed to cool to room temperature and then stored in bottles under nitrogen.

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A run was made by loading the reactor with a 20 gram char sample. The reactor was purged with helium and the temperature was raised to the desired level. At that point the pressure in the reactor was raised to operating conditions by manually injecting water through the pump. When the run pressure was achieved, the pump was set in the automatic mode. If syngas was used, the supplementary gas valve was also opened at the start of the run. Steam and syngas (if used) were then fed to the reactor. At the end of a run, the feed was shut off and the unit depressured.

During the run, gas analyses and cumulative dry gas volumes were obtained. From this data the carbon gasified is calculated. Assuming that the oxygen content of the char is small in relation to the oxygen content of the steam fed, the steam conversion is obtained from the oxygen content of the dry product gases.

Runs were made in the fixed bed reactor with Illinois coal catalyzed with 10% and 20% K_2CO_3 with steam as the gasifying medium. Temperatures of 1200°F and 1300°F were used and pressures varied from 0 to 500 psig. Steam flows ranged from 3 to 100 gm/hr. With these conditions, steam conversions from 10% to 80% and total carbon conversions from 50% to 100% were obtained. Material balances on hydrogen were used to check the consistency of the data. The balance closures ranged from 100% to 105% for typical runs.

Results

During the runs it was observed that the steam gasification rate was independent of pressure. The gasification rate was found to increase with an increasing rate of steam fed to the reactor. Additionally, at high steam flow rates, or low steam conversions, the gasification rate was directly proportional to the catalyst loading. One explanation for these observations is that the kinetics are controlled by a strong product inhibition. This suggests that a kinetic expression in the classical Langmuir-Hinshelwood form may be used to fit the data. It was further seen that methane and carbon dioxide were in chemical equilibrium with the other gas phase components for the conditions studied, i.e., the methanation and shift reactions are at equilibrium.

DATA INTERPRETATION

Fixed Bed Reactor Model

A mathematical model for the fixed bed reactor was developed based upon the observed behavior. Plug flow of gas through the bed is assumed. It is also assumed that strong product inhibition results in a high rate of gasification over a very short distance of the bed followed by a slower rate over the remaining length of the bed where higher partial pressures of products exist. This assumption leads to a simplified picture for the fixed bed reactor shown in Figure 3. In this model the reaction proceeds so as to form a sharp "carbon burnoff front." If little or no carbon is present, gasification will not take place. Therefore, the potassium catalyst which is left behind this "burnoff front" does not contribute to the reaction rate.

The equation describing conversion in the plug flow reactor is

$$\int_0^V \frac{dV}{N^\circ_{H_2O}} = \int_0^x \frac{dx}{-r_G} \tag{1}$$

where $\underline{V}$ is the reactor volume, $N^\circ_{H_2O}$ is the molar rate of steam fed to the reactor, r_G is the molar rate of the carbon-steam gasification reaction per unit volume and $\underline{x}$ is the extent of reaction defined as moles carbon gasified per mole steam fed. The sharp burnoff front model provides a relationship between the carbon remaining in the bed and the effective fixed bed reactor volume,

$$n_C = C_C \, V \tag{2}$$

where n_C is the instantaneous moles of carbon in the bed, $\underline{V}$ is the effective reactor volume, and C_C is the proportionality constant with the dimension moles carbon per unit volume. Based upon initial bed conditions C_C will have a value of approximately 0.045 gmole/cc. Substitution of Equation (2) into Equation (1) provides

$$\frac{n_C}{N^\circ_{H_2O} \, C_C} = \int_0^x \frac{dx}{-r_G} \tag{3}$$

This model may now be used for the identification of acceptable forms for the rate, r_G, and to obtain best fit values for the parameters in these expressions.

A Langmuir-Hinshelwood type expression for heterogeneous catalytic kinetics as applied to the carbon-steam reaction may be written in the generalized form.

$$r_G = \frac{k[p_{H_2O} - p_{CO} \, p_{H_2}/K_G]}{1 + \sum_i (b_i p_i + \sum_j b_{ij} p_i \, p_j)} \tag{4}$$

where p_{H_2O}, p_{CO}, p_{H_2}, etc. represent the partial pressures of these components, $\underline{k}$ is the kinetic rate constant for the carbon-steam reaction, K_G

is the equilibrium constant for this reaction, and the b's represent the adsorption constants, no more than four of which will be allowed to be nonzero in any one model being tested.

Equation (4) when substituted into Equation (3) gives

$$\theta = \frac{n_c}{N^{\circ}_{H_2O}} = \frac{C_c}{k}\int_0^x \frac{dx}{R_G} + \sum_i \frac{b_i C_c}{k}\int_0^x \frac{p_i}{R_G}\,dx + \sum_i \sum_j \frac{b_{ij} C_c}{k}\int_0^x \frac{p_i p_j}{R_G}\,dx \qquad (5)$$

where the reaction driving force term in the denominator of each of the integrals is given by

$$R_G = -[p_{H_2O} - p_{CO}p_{H_2}/K_G] \qquad (6)$$

For a given conversion, the shift and methanation equilibrium relationships are sufficient to calculate the partial pressures of all components (H_2, CO, CH_4, CO_2, H_2O) in the gas phase. Using a closely spaced series of incremental values for x, the partial pressures were accurately mapped over a range of conversions. This needed to be done only once. These partial pressures were then substituted as required into the expressions under the integrals shown in Equation (5). The values of these integrals for any specified conversion are then obtained by a Simpson's rule numerical integration of the expression under the integrals.

The data collected in the fixed bed steam gasification experiments described above were used to calculate and tabulate conversion, x, moles carbon gasified per mole steam fed as a function of holding time, θ, moles instantaneous bed carbon per molar steam flow rate. The "carbon burnoff front" model for fixed bed potassium catalyst gasification requires that the data for x as a function of θ collected for different steam flow rates must all mesh together to give a single curve for any fixed temperature, pressure, and catalyst loading. A plot of data collected for steam gasification over a range of steam flowrates at 1300°F, 500 psig and 20% K_2CO_3 on Illinois coal is provided in Figure 4. For each experimental run the initial data points are at the right and move to the left as carbon is depleted from the bed. The flat region in the data at the upper right of Figure 4 represents the equilibrium limit for the carbon steam reaction. This limit corresponds to a carbon activity of about twice that of β-graphite. The region at the lower left of the diagram shows the carbon conversions limited by the rate of reaction. The data points at the different steam rates overlap in the required manner over three orders of magnitude of residence time. Thus, the experimental observations are consistent with the postulated model. This reactor model was then used as the basis for the analysis of the reaction data.

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Parameter Estimation

The coefficients in front of the integrals in a series of particular forms of Equation (5) were estimated by regression analysis. The regression data base used consisted of the results of the steam gasification runs at 500 psig described above as well as runs at 0, 100 and 250 psig at steam rates of 6, 12 and 24 gm H_2O/hr. Two additional series of runs were conducted at 500 psig and the same three steam rates. The first was at 1200°F and 20% K_2CO_3 and the second was at 1300°F and 10% K_2CO_3. The data from these runs were used to assess the effect of temperature and catalyst loading on gasification rate.

Numerous kinetic models were formulated and tested by regression for the constants in Equation (5). These models consisted of all combinations of from one to four terms involving the partial pressures of H_2, CO, and H_2O and the cross products of the partial pressures of H_2 and CO, and H_2 and H_2O. Those which gave negative coefficients on regression were discarded as being physically unreal. Four additional models were discarded because they gave an infinite rate in the limit of zero steam conversion. The three models which remained are

(A)
$$r_G = \frac{k(p_{H_2O} - p_{CO}\ p_{H_2}/K_G)}{p_{H_2} + b_1\ p_{H_2O}} \tag{7}$$

(B)
$$r_G = \frac{k(p_{H_2O} - p_{CO}\ p_{H_2}/K_G)}{p_{H_2} + b_1\ p_{H_2}\ p_{CO} + b_2 p_{H_2O}} \tag{8}$$

(C)
$$r_G = \frac{k(p_{H_2O} - p_{CO}\ p_{H_2}/K_G)}{p_{H_2} + b_1\ p_{CO} + b_2\ p_{H_2O}} \tag{9}$$

All are independent of pressure. The variance of the residuals around the regression line for these are A: 0.0556, B: 0.0519, and C: 0.0562. Since Model B has a smaller variance than A or C, it was chosen as the basis for further analysis. However, further studies should be done to better discriminate between these and possibly other kinetic expressions. The coefficients obtained by regression of Model B are

$$\frac{C_c}{k} = 1.603\ hr$$

$$\frac{b_1 C_c}{k} = 0.3371\ hr/atm$$

$$\frac{b_2 C_c}{k} = 0.0954\ hr$$

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These coefficients were used in Equation (5) to compute the values of θ required to achieve the various measured conversion levels. These calculated values are compared to the actual holding times in Figure 5. While there is scatter to the data, it is seen that the model provides a reasonable fit over the broad range of pressures (0-500 psig) and flowrates (3-100 gm/hr) considered.

Using the approximate value of $C_c = 0.045$ gmole/cc, the values for the parameters at 1300°F and 20% K_2CO_3 loading may be expressed as

$$k = 0.0281 \ \frac{\text{gmole C}}{\text{hr·cc}}$$

$$b_1 = 0.210 \ \text{atm}^{-1}$$

$$b_2 = 0.0595$$

It was found by comparing the 1200°F and 1300°F data that the rate constant, k, has an activation energy of 30 kcal/gmole in the Arrhenius expression. Furthermore, its value at the 10% K_2CO_3 loading was approximately half that at the 20% K_2CO_3 level. Hence, within this range k may be expressed as

$$k = k_0 C_K \exp(- E/RT). \tag{10}$$

where k_0 is the frequency factor, C_K is the moles of catalytically active potassium per unit volume, E is the activation energy, R is the universal gas constant and T is the absolute temperature. For 20% K_2CO_3 on Illinois coal the value of C_K for the fixed bed of char is typically

$$C_K = 0.0021 \ \text{gmole/cc}$$

On this basis the value of the frequency factor may be computed as

$$k_0 = 6.80 \times 10^7 \ \text{gmole C/hr·gmole K}$$

for

$$E = 30 \ \text{kcal/gmole.}$$

The ratio of holding times necessary to attain a given conversion level, x, at two different temperatures and catalyst levels is given by

$$\frac{\theta_1}{\theta_2} = \frac{k_2}{k_1} = \frac{C_{K_2}}{C_K} \quad \exp\left[- \frac{E}{R}\left(\frac{1}{T_2} - \frac{1}{T_1}\right)\right] \tag{11}$$

This assumes that the temperature difference does not significantly affect the equilibrium calculation for the partial pressures. Equation (11) allows the definition of an "equivalent residence time," $\underline{\theta}^*$, which can be used to combine data collected at different temperatures and catalyst levels. The quantity $\underline{\theta}^*$ is defined as the holding time at $\underline{T}^*$ and C_K^* which will give the same conversion as that obtained with a holding time $\underline{\theta}$ at temperature $\underline{T}$ and catalyst concentration $C_{\underline{K}}$. Specifically,

$$\theta^* = \theta \; \frac{C_K}{C_K^*} \; \exp\left[-\frac{E}{R}\left(\frac{1}{T} - \frac{1}{T^*}\right)\right] \tag{12}$$

This relationship was tested for its ability to correlate 500 psig fixed bed reaction data collected at 10% K_2CO_3-1300°F and 20% K_2CO_3-1200°F with the data base collected at 20% K_2CO_3-1300°F. The result is given by the data points shown in Figure 6 where conversion, $\underline{x}$, is plotted as a function equivalent residence time, $\underline{\theta}^*$, with all data adjusted if needed to 1300°F and 20% K_2CO_3. It is seen that the data appear uniformly correlated by this expression.

Generalized Fixed Bed Model

The above kinetic relationships apply to a pure steam feed. In order to apply them to the synthesis gas recycle case, they must be generalized for mixed gas input to the fixed bed. This may be done by writing the differential equations describing the molar flow of each molecular species through the bed and numerically integrating these over the effective volume. These equations are

$$\frac{d \, N_{H_2}}{dz} = A \; (-3 \, r_M + r_S + r_G) \tag{13}$$

$$\frac{d \, N_{CO}}{dz} = A \; (-r_M - r_S + r_G) \tag{14}$$

$$\frac{d \, N_{CH_4}}{dz} = A \; r_M \tag{15}$$

$$\frac{d \, N_{CO_2}}{dz} = A \; r_S \tag{16}$$

$$\frac{d \, N_{H_2O}}{dz} = A \; (r_M - r_S - r_G) \tag{17}$$

where $N_{\underline{i}}$ is the molar flow rate of component $\underline{i}$, $\underline{z}$ is the distance down the bed, $\underline{A}$ is the cross-sectional area of the bed, and $\underline{r_M}$, $\underline{r_S}$ and $\underline{r_G}$ are the

rates of the methanation, shift, and carbon-steam gasification reactions respectively expressed as moles per unit reactor volume per unit time.

The reaction rate expressions used for the shift and methanation reactions are

$$r_S = k_S (p_{CO} \ p_{H_2O} - p_{CO_2} \ p_{H_2}/K_S) \tag{18}$$

$$r_M = k_M (p_{CO} \ p_{H_2}^3 - p_{CH_4} \ p_{H_2O}/K) \tag{19}$$

where k_S and k_M are the respective rate constants and K_S and K_M are the respective equilibrium constants. These reactions may be forced to equilibrium by assigning arbitrarily large rate constants. The reaction rate expression used for the potassium catalyzed carbon-steam reaction is obtained by combining Equations (8) and (10)

$$r_G = \frac{k_o \ C_K \ exp(-E/RT) \ [p_{H_2O} - p_{CO} \ p_{H_2}/K_G]}{p_{H_2} + b_1 \ p_{CO} \ p_{H_2} + b_2 \ p_{H_2O}} \tag{20}$$

The ordinary differential Equations (13)-(17) were numerically integrated by a Runga-Kutta-Fehlberg procedure for a series of cases considering pure steam fed to a fixed bed reactor at 500 psig, 1300°F and $C_K = .0021$ gmoles potassium per cc (corresponding to 20% K_2CO_3 on Illinois coal). The conversion, $\underline{x}$, was determined at various distances, z, down the bed from

$$x = \frac{N_{CO} + N_{CH_4} + N_{CO_2}}{\overset{o}{N}_{H_2O}} \tag{21}$$

The residence time corresponding to each conversion was computed as

$$\theta = \frac{C_c \ A \ z}{\overset{o}{N}_{H_2O}} \tag{22}$$

The integrations performed in this manner for various steam flowrates overlapped to give the single correlation line shown in Figure 6. This line is seen to provide a reasonable fit to the data.

Model Verification Experiments

To test the predictive capability of the kinetic model with a mixed gas feed, two fixed bed gasification runs were made with steam plus syngas (H_2 + CO)

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at 1300°F. One run was made with 5 liter per hour syngas at 500 psig. The
second was made with 15 liter per hour syngas at 100 psig. Both runs were
made with 12 grams per hour steam feed. In both cases the syngas composition
was 75 mole % H2 and 25 mole % CO. In these experiments the conversion, $\underline{x}$,
was computed as

$$x = \frac{N_{CO} + N_{CH_4} + N_{CO_2} - \overset{\circ}{N}_{CO}}{\overset{\circ}{N}_{H_2O}} \tag{23}$$

where $\overset{\circ}{N}_{CO}$ is the molar rate of carbon monoxide fed to the reactor. The
residence time is computed by Equation (22). A comparison between the predicted
and experimental conversions for these two experiments is shown in Figure 7.
Good agreement is observed in the 500 psig case. The conversions obtained here
are essentially the same as observed above for pure steam feed. At 100 psig with
higher syngas flow, the data show a lower conversion than at 500 psig for the
same residence time. It is also seen that the model underpredicts the actual
conversion. This may be due, in part, to the use of parameters which are
derived from pure steam data.

CONCLUSIONS

An empirical Langmuir-Hinshelwood kinetic model for the potassium catalyzed
gasification of Illinois #6 bituminous coal has been developed. This model
provides a good fit to fixed bed reactor data over pressures ranging from
atmospheric to 500 psig and a 30-fold range of steam flow rates. It also
predicts conversions for the temperature range 1200°F to 1300°F and catalyst
loadings from 0.1 to 0.2 grams K_2CO_3 per gram of coal. For the catalyst
levels examined, the gasification rate was proportional to the amount of catalyst
present. Additional studies need to be performed over a broader range of
catalyst loadings to determine the limits of this relationship. It was also
shown that these kinetics can be applied to predict trends in conversion for
H_2O, H_2 and CO mixed gas feeds.

The kinetic expression obtained has been shown to have adequate predictive
capabilities in the range of interest. It is in a form which can be used
directly in the development of models for fluid bed gasification reactors.
Thus, the goal for this study has been achieved. Future work will be directed
toward formulating a fluid bed reactor model.

ACKNOWLEDGEMENT

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Nomenclature

A cross-sectional area of reactor

b adsorption constant in Langmuir-Hinshelwood rate expression

C_C carbon concentration, moles C per unit reactor volume

C_K potassium concentration, moles K per unit reactor volume

E activation energy in Arrhenius expression for carbon-steam reaction rate constant

k rate constant for carbon-steam reaction

k_o frequency factor in Arrhenius expression for carbon-steam reaction rate constant

k_M rate constant for methanation reaction

k_S rate constant for shift reaction

K_G equilibrium constant for carbon-steam reaction, atm

K_M equilibrium constant for methanation reaction, atm^{-2}

K_S equilibrium constant for shift reaction

N_i molar flow rate of component $\underline{i}$

N_i^o molar flow rate of component $\underline{i}$ fed to reactor

n_C moles carbon (total in reactor)

p_i partial pressure of component $\underline{i}$, atm

R universal gas constant

R_G driving force for carbon-steam reaction, see Equation (6)

r_G molar rate of carbon-steam reaction per unit reactor volume

r_M molar rate of methanation reaction per unit reactor volume

r_S molar rate of shift reaction per unit reactor volume

V volume of fixed bed reactor

x extent of reaction, moles carbon reacted per mole H_2O fed

z distance from start of fixed bed reactor

θ residence time in fixed bed, moles bed carbon-hr/mole H_2O fed

References

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FIGURE 1. OVERALL FLOW PLAN OF THE EXXON CATALYTIC COAL GASIFICATION PROCESS

78B-6-5-1

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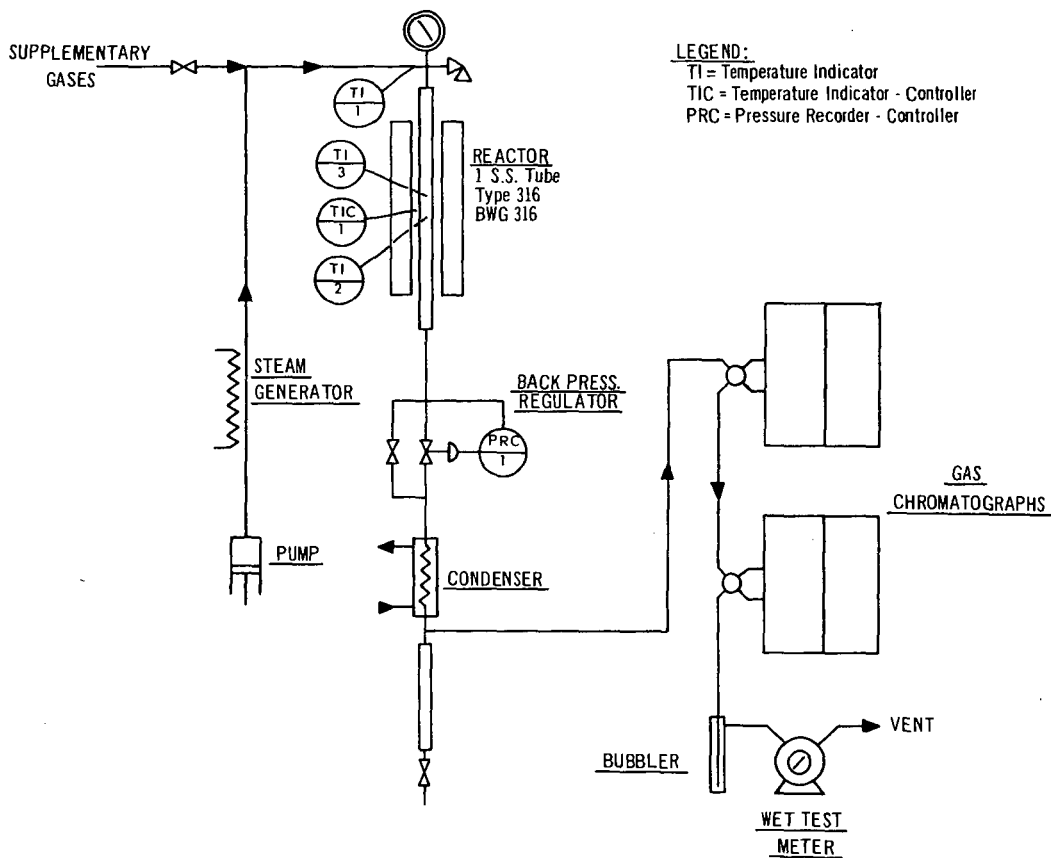
FIGURE 2   SCHEMATIC DIAGRAM OF BENCH SCALE
FIXED BED GASIFICATION UNIT

78B-6-4-8

Fixed Bed at
Start of Run

Steam →

$Z = 0$
$Z = 0.3L$
{ If Gas is "Sampled" here, it can be Envisioned as Being the Same as The Effluent Coming From the Bed on the Right

$Z = L$

Reacted
Effluent

100% of
Initial
Carbon
and K

Fixed Bed at
Time = t

Steam →

Ash Particles with no Carbon (Containing 70% of Initial K

Char Particles with 30% of Initial Carbon and K

$Z = 0$
$Z = 0.3L$
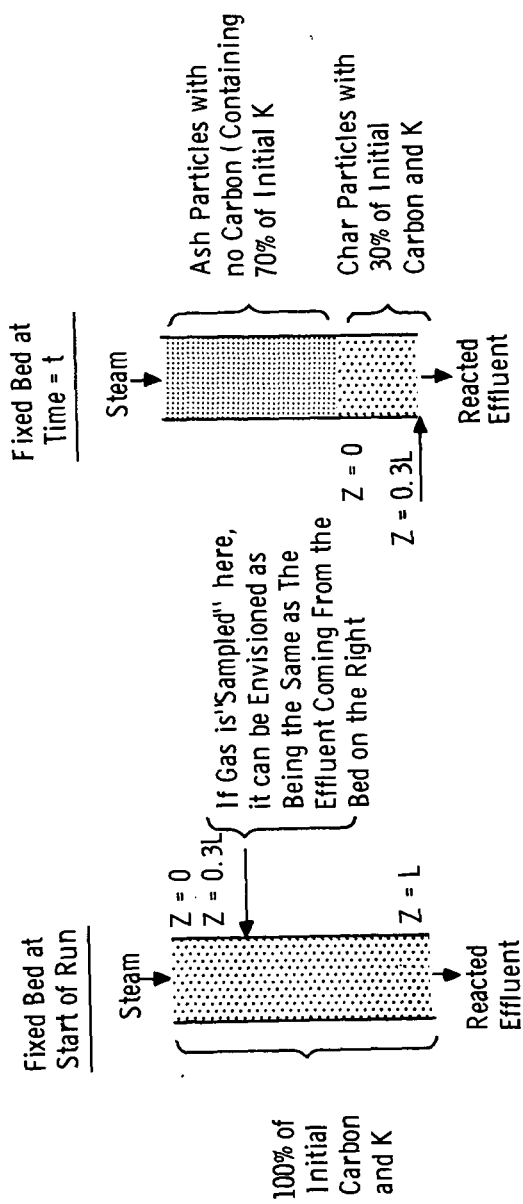
Reacted
Effluent

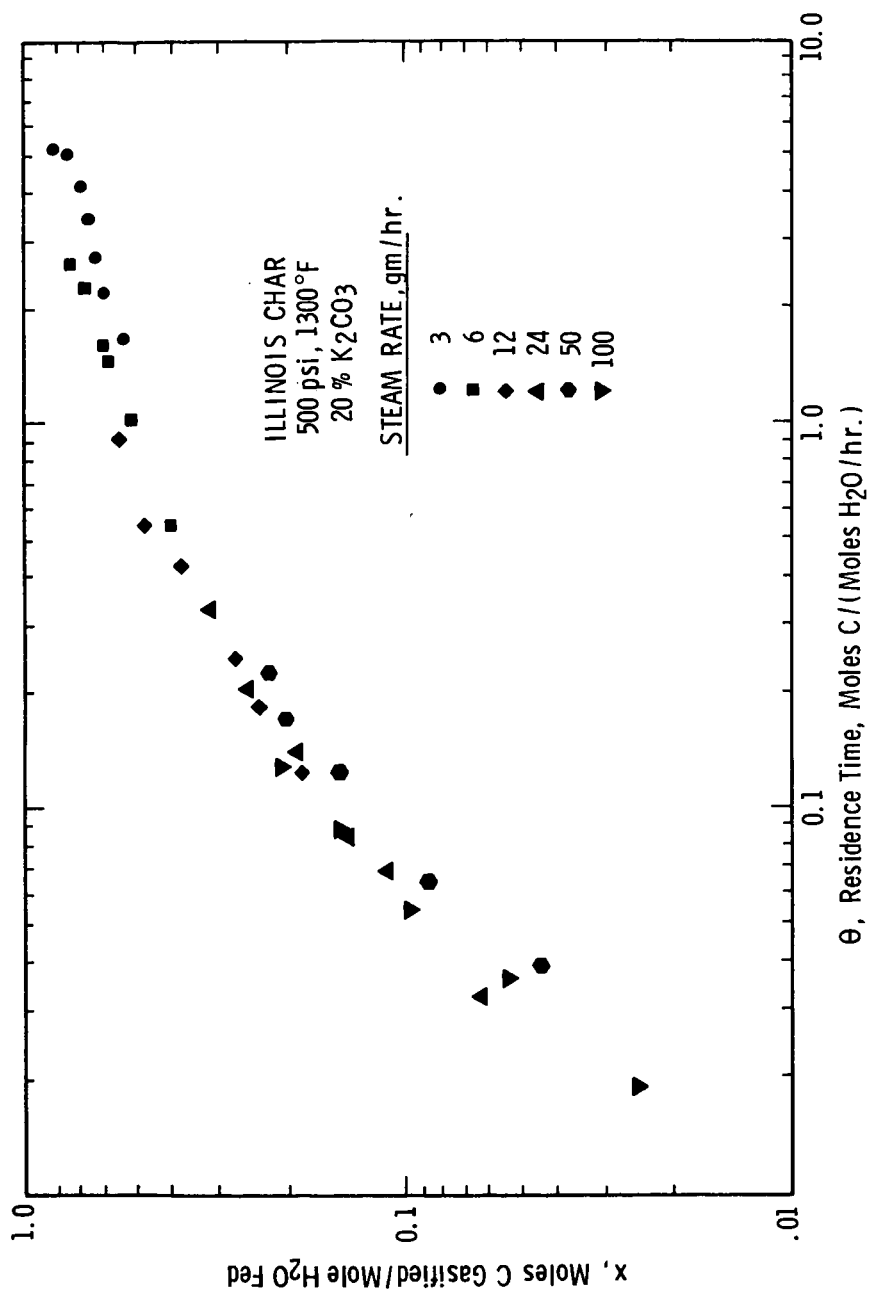FIGURE 3. SIMPLIFIED MODEL FOR FIXED BED REACTOR IN POTASSIUM CATALYZED STEAM GASIFICATION

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FIGURE 4   EXTENT OF CARBON STEAM REACTION AS A
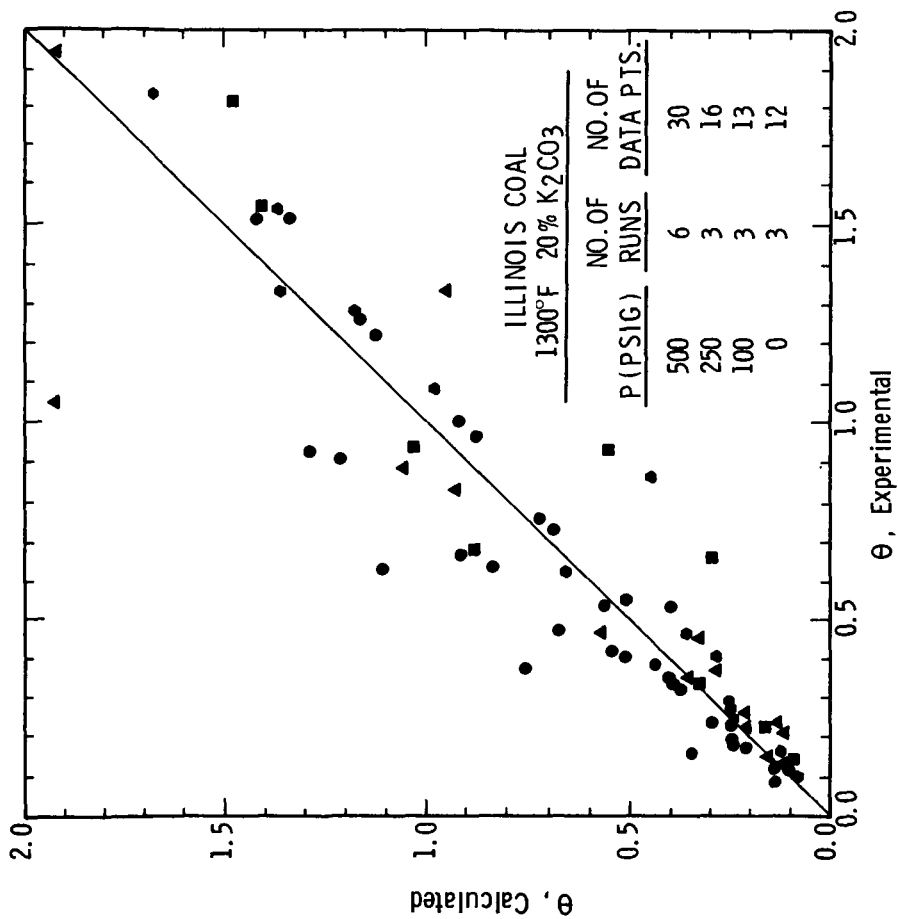FUNCTION OF RESIDENCE TIME

78B-6-4-10

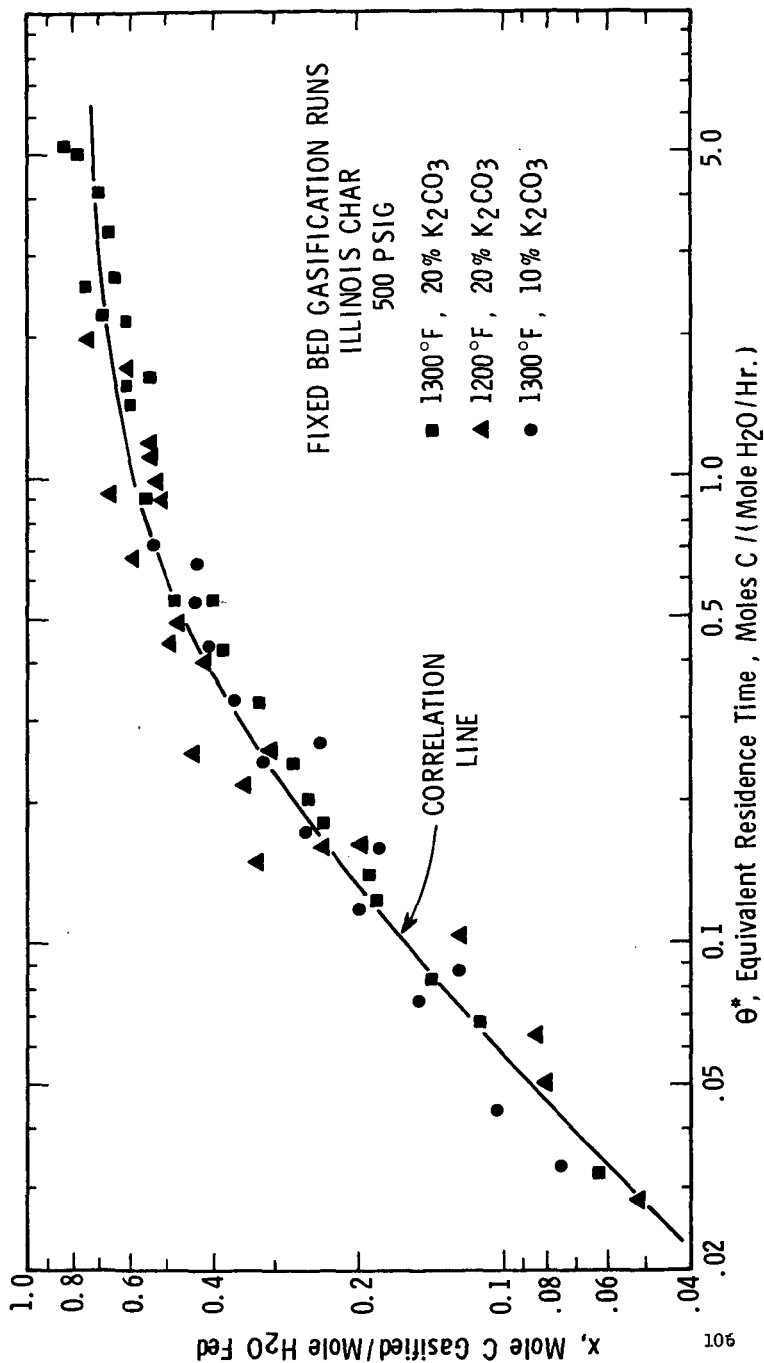FIGURE 5 COMPARISON OF EXPERIMENTAL AND PREDICTED HOLDING TIME, θ , TO ATTAIN A GIVEN CONVERSION FOR MODEL "B"

78B-6-4-11

FIXED BED GASIFICATION RUNS
ILLINOIS CHAR
500 PSIG

- ■ 1300°F, 20% K_2CO_3
- ▲ 1200°F, 20% K_2CO_3
- ● 1300°F, 10% K_2CO_3

CORRELATION
LINE

θ^*, Equivalent Residence Time, Moles C /(Mole H_2O/Hr.)

x, Mole C Gasified/Mole H_2O Fed

FIGURE 6   COMPARISON OF FIXED BED DATA WITH MODEL PREDICTION
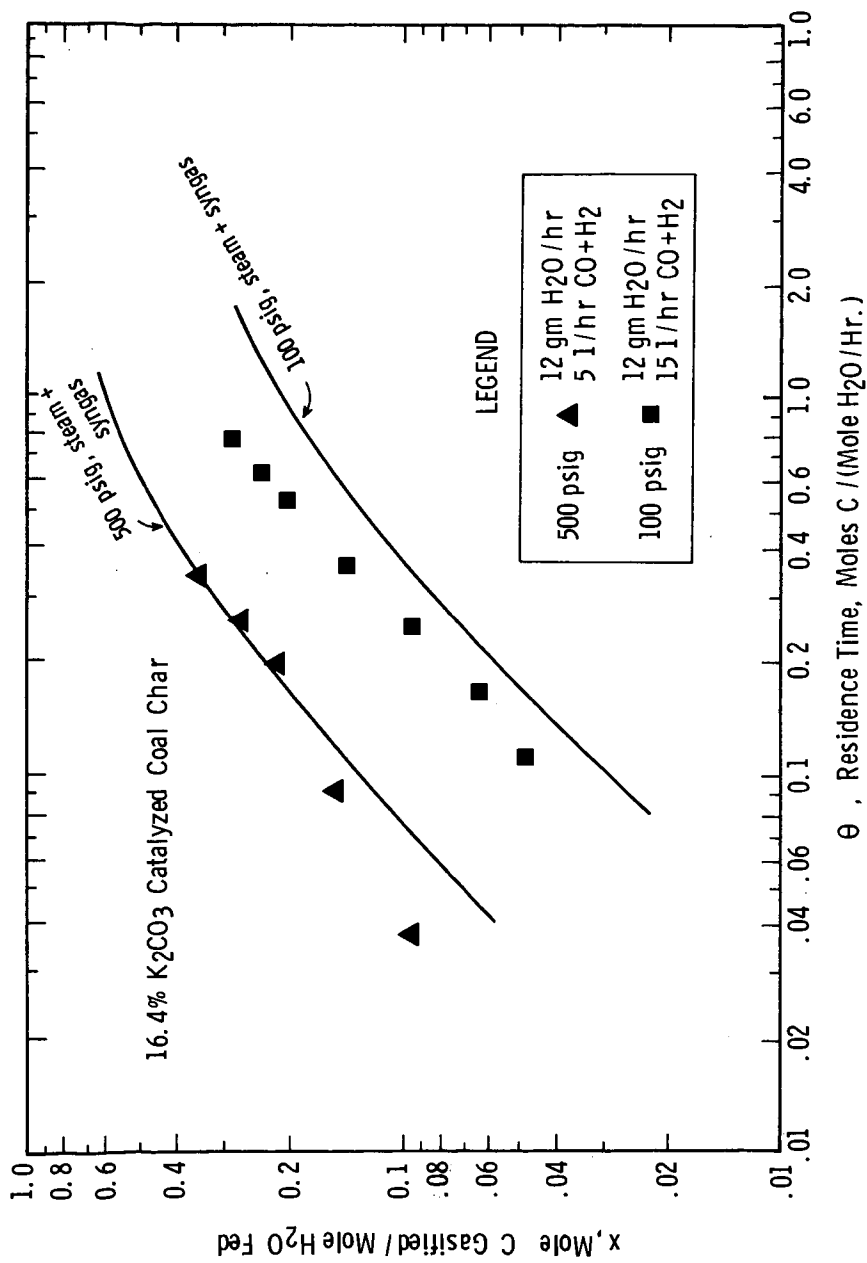FOR STEAM FEED

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78B-6-4-12

FIGURE 7   COMPARISON OF FIXED BED DATA WITH MODEL PREDICTIONS
FOR STEAM/SYNGAS FEED

78B-6-4-13

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